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STRUCTURAL APPLICATION OF HIGH STRENGTH,
HIGH TEMPERATURE CERAMICS

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ABSTRACT

The operation of rocket engine turbine pumps is limited by the temperature restrictions of metallic components used in the systems. Mechanical strength and stability of these metallic components decrease drastically at elevated temperatures. Ceramic materials that retain high strength at high temperatures appear to be a feasible alternate material for use in the hot end of the turbopumps. This project identified and defined the processing parameters that affected the properties of Si_3N_4 , one of the candidate ceramic materials. Apparatus was assembled and put into operation to hot press Si_3N_4 powders into bulk material for in house evaluation. A work statement was completed to seek outside contract services to design, manufacture, and evaluate Si_3N_4 components in the service environments that exists in SSME turbopumps.

INTRODUCTION

The Space Shuttle Orbiter is the heart of NASA's space transportation system during the decade of the eighties. This reusable vehicle trims the cost of space travel while increasing NASA's capabilities in space. The system can carry a crew of three, four scientists, and a sixty five thousand pound load into orbit. This Orbiter returns to earth like an airplane and it is anticipated that it can make one hundred round trips into space and back. Although the Shuttle System utilizes the most advanced aerospace technology available to date to achieve economical and useful space flight, it is anticipated various improvements will be made on the system as experience is gained and new technologies emerge. Marshall Space Flight Center has the main responsibility for the Space Shuttle main engine and solid rocket boosters used in this system.

One area in which improvement is desired is in the high-pressure turbopumps, both fuel and oxidizer, in the Space Shuttle main engine. The operation of these pumps is limited by temperature restrictions of the metallic components used in these pumps. Spot melting, oxidation, and erosion-corrosion are some of the problems encountered in the turbopumps.

High strength refractory ceramics are being considered for use as structural materials in these turbopumps. This will alleviate the problems mentioned above and in addition permit a several hundred degree fahrenheit increase in operational temperatures. This increase in temperature will increase the overall efficiency of the space shuttle main engines, which in turn will permit a larger payload. This ceramic material which appears to have the best possibility of being utilized is silicon nitride, Si_3N_4 .

OBJECTIVES

The objectives of this two year study were:

1. Identify and define the processing parameters that affect the properties of Si_3N_4 ceramic materials.
2. Design and assemble equipment required for processing high strength ceramic.
3. Design and assemble test apparatus for evaluating the high temperature properties Si_3N_4 .
4. Conduct a research program of manufacturing and evaluating Si_3N_4 materials as applicable to rocket engine applications.

ENGINEERING ANALYSIS

Silicon nitride has the desired properties for utilization as a high temperature structural material. These properties include high strength, low coefficient of friction, high decomposition temperature, good corrosion resistance, good oxidation resistance, high wear resistance and good thermal shock resistance. These positive features have been known for two decades and slowly silicon nitride is approaching the fulfillment of its potential. Two problems have slowed the use of silicon nitride. The first is the fabricating of suitable or useful shapes with the desirable properties. The second is the brittleness of silicon nitride, and indeed, all ceramic materials. Traditionally, all engineering design compromises on the selection of a material to use in an application, taking into consideration the total cost of a material and the use properties of that material. The evaluation of these compromises in design generally show that a metallic material has a higher net profit. Because of this, there is very little design experience with structural applications of silicon nitride or other brittle ceramics. It is being considered at this time because of the great need for the potential of Si_3N_4 and the development of computer capability to define the stresses as required for brittle material design.

PROCESSING PARAMETERS

There are three basic methods of forming bulk Si_3N_4 . The first of these three is called "reaction-bonded" Si_3N_4 whereby formed piece of pressed silicon powders is nitrided in nitrogen gas in the range of 1300-1400°C. During this nitridation process, the silicon powders are reacted with N_2 gas to form a mixture of α - and β - Si_3N_4 . The second method is called "hot-pressed" Si_3N_4 . This process entails the nitriding of Si powders to form α - Si_3N_4 powders. These Si_3N_4 powders are mixed with desirable additives and then pressed into a compact in a graphite mold, under a pressure of 1-2 tons/in², at a temperature of 1700-1800°C. This results in a high strength, high density β - Si_3N_4 product. The third process is called "sintered" Si_3N_4 . In the sintered product, α - and/or β Si_3N_4 powders are mixed with desirable additives and then pressed into the desired shape. This pressed bulk is then sintered in a controlled

atmosphere at 1800-1911°C for a considerable length of time. The product of this sintering operation is essentially β -Si₃N₄. Reaction bonded and sintered silicon nitride generally increases in strength with an increase in use temperature up to the 1400°C range and then falls off. Hot pressed silicon nitride is strongest at room temperature and the strength falls off gradually until the 1200°C range and then falls off more rapid. Since reaction bonded and sintered silicon nitride both have lower room temperature strength than hot pressed there is considerable overlap of strengths in the 1200°C to 1450°C range. The exact strength and other properties in this range is very dependent on the starting materials and other processing parameters, such as the specific sintering aid utilized.

In addition to the above parameters, there are two hexagonal crystal structures of silicon nitride, α -Si₃N₄ and β -Si₃N₄, with the α -Structure unit cell being approximately twice the size as the β -Structure unit cell. Since either structure can be produced from the other by rotation of two basal planes, requiring breaking and forming primary bonds, the α - β transformation requires time and energy. Two possible methods are solutioning-precipitation and volatilization-condensation. Both structures can be formed over a wide range of temperatures and both are relatively stable, although β -Si₃N₄ appears to be more stable at temperatures in excess of 1500°C. The formation of Si₃N₄ through a vapor-phase reaction favors the formation of the α -Structure, while precipitation from the liquid state favours the β -Structure formation. The morphology of the formed crystals is important with the fibrous β -Structure preferred because it enhances the strength and toughness of the resulting bulk material.

The processing parameters that are controlled to obtain the desired properties in the silicon nitride include:

- a. Initial particle size and size distribution
- b. Impurities
- c. Milling and Mixing procedures
- d. Sintering aids
- e. Reaction (sintering) temperature
- f. Reaction (sintering) pressure
- g. Reaction (sintering) time
- h. Reaction (sintering) atmosphere
- i. Post heat treatment

Not all of these parameters apply to each of the three types of bulk solid Si₃N₄. Normal impurities present in Si₃N₄ regardless of type are iron, aluminum, calcium, magnesium and oxygen. These elements are usually present in the combined state such as oxides, silicides or silicates.

An excellent article on how all of the above variables affect the final physical properties of the silicon nitride is given in the paper by Larsen et al (Ref. 1) which was presented in 1981.

EQUIPMENT ASSEMBLY

Equipment was assembled and checked out to hot press silicon nitride powders into a bulk shape. The equipment utilized was as follows:

Lepel High Frequency Induction Furnace, model T-20-3-KC-E-H, 45 KVA, 20 KW Radio Frequency Output, 180-450 KC Frequency range, water cooled at a rate of ten gallons per minute.

Corning Precision Deviation Controller model 8810.

Leeds and Northrup Automatic Optical Pyrometer.

BLH Electronics Universal Transducer Indicator, BLH model 350, 25,000 pound load cell at 100%.

Graphite Dies, 1½ inch diameter.

Cavity, dual plungers 1½ diameter and 4 inches in length, outside shell 3½ inch diameter and a height of 6 inches.

Silicon nitride powders, Cerac Pure, Cerac Incorporated, Milwaukee, Wisconsin, -325 mesh, mixture of alpha and beta. Phases, about 2 micron average diameter, 99.9% pure, nitrogen content 38%.

Several runs were completed utilizing this equipment with the final bulk density of the silicon nitride being 99.4% of theoretical. Two problem areas requiring additional effort are:

The sintered silicon nitride bonds to the plungers during the sintering process. It is suggested that small sacrificial dish be inserted between the powders and the plungers.

Water pressure variations causes the safety switch to activate cutting off the Induction Furnace during low pressure periods. Larger lines, or a large surge tank should alleviate this problem.

DESIGN, MANUFACTURING AND EVALUATION OF Si_3N_4 AS A STRUCTURAL COMPONENT OF THE SSME

This portion of the objectives of this research project was beyond the scope of the ASEE-NASA program, and a request for proposal was prepared. The work statement of the RFP follows.

General

It is requested that a contract be negotiated to define the parameters affecting the use of high temperature, high strength ceramics as structural components of the Space Shuttle Main Engine. This contract should be negotiated on a noncompetitive basis and awarded as a cost-plus fixed fee contract.

Background

Operation of the Space Shuttle on a more efficient basis of providing more thrust without additional fuel consumption should directly result in an increase in payload capacity. One means of achieving this increased efficiency is to increase the operational temperature of the turbopumps. The operational temperatures of the turbopumps are presently limited by the capabilities of the superalloys in the turbopump. The use of structural ceramic materials should permit operation of the turbopumps up to 2500° F.

Several materials systems are being developed that may provide the properties necessary for the higher temperature operation. Silicon nitride, silicon carbide, transformation toughened zirconia, and fiber reinforced ceramics appear promising as candidate materials systems for structural applications. The material for this application must possess the properties of high strength at elevated temperatures, good fracture toughness, and thermal shock resistance.

Work Statement

Phase I - Environment Definition

Definition of the environment that the ceramic components of a turbopump will be exposed to will acutely effect the material, processing, and design of the components. The contractor shall define in detail the environments in the turbopumps during start-up, operation, and shut down. The environmental parameters of interest are as follows:

1. Temperature Extremes
2. Rate of Temperature Change
3. Heat Flux Extremes
4. Rate of Change of Heat Flux
5. Gas Mass Flow
6. Gas Composition
7. Combustion Products Composition
8. Change in Gas Composition Ratios
9. Uniformity of Gas Composition Relative to Location
10. Temperature Profile
11. Stress Levels
12. Rotational Speed
13. Pressure Extremes
14. Rate of Change of Pressure
15. Other Stress Sources and their levels
16. Any Other Parameter that would Affect

This data shall be compiled and be available for use in the following phases of this program.

Phase II - Components Study

Based on the data generated in Phase I and knowledge of structural ceramics, the contractor shall conduct a study to identify the components that appear to be candidates for being fabricated from high strength refractory ceramic materials. The rationale utilized to reach these

conclusions shall be provided. Acceptable design criteria such as Wiebull Modulus or other accepted techniques shall be utilized to establish an acceptable degree of reliability for the candidate structures/materials.

Phase III - Design Parameters

The contractor shall apply the results of Phase I and Phase II of this work statement to identify and quantify the critical design parameters of selected components. Included should be the area of utilizing brittle materials as a structural member, where some attention given to:

1. Method of attachment of components
2. Method of joining of components
3. Design concepts to eliminate point contacts
4. Design concepts to maximize compressive loading of ceramics and minimize other types of loading
5. Design concepts to minimize thermal shock effects
6. Design concepts to minimize impact potential and effect

The contractor will also identify any other potential problem areas expected to be encountered and suggest design concepts to minimize these problems. This phase of the project will require the expertise of experienced design personnel in the field of utilization of brittle materials as structural members.

Phase IV - Design Variables

The contractor upon completion of Phase III shall supply a format for the preparation of ceramic components for the turbopumps to be utilized in the Space Shuttle Main Engine. This format shall contain, but not limited to, the following:

1. The selected shapes of the components
2. Materials characterization
 - a. Starting Powders
 1. size
 2. shape
 3. purity
 - b. Required alpha or beta Phase Ratio
 - c. Suppliers
3. Processes
 - a. Forming
Sintering
Final Shaping Tolerance
4. Attachment Techniques

5. Special Handling Required

6. Required Specifications of as Completed Components

This phase will require the collaboration of design personnel and material personnel.

CONCLUSIONS AND RECOMMENDATION:

Si_3N_4 is a viable material candidate for utilization in high temperature structural applications. However, it must be fully evaluated under use conditions and environments before utilization. Main areas of concern are thermal shock, methods of attachment, and reactive atmosphere when considering application in rocket engine turbine. It is recommended that these areas be studied and evaluated under actual environments existing in the turpumps early in the program. These evaluation should continue throught the life of the program as long as any change is made in the Si_3N_4 processing parameters. Additional studies could include:

- a. Creep and strength improvement based on different sintering aids
- b. Forming techniques
- c. New processing techniques such as reaction-bonding followed by sintering
- d. Basic studies on the glass bond formed during sintering

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